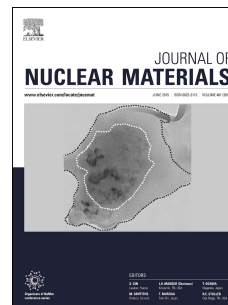


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Raman spectroscopy analysis of air grown oxide scale developed on pure zirconium substrate.

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Using Raman spectroscopy technique, external and internal parts of zirconia oxide films developed at 500°C and 600°C on pure zirconium substrate under air at normal atmospheric pressure have been examined. Comparison of Raman peak positions of tetragonal and monoclinic zirconia phases, recorded during the oxide growth at elevated temperature, and after cooling at room temperature have been presented. Subsequently, Raman peak positions (or shifts) were interpreted in relation with the stress evolution in the growing zirconia scale, especially closed to the metal/oxide interface, where the influence of compressive stress in the oxide is the biggest. Reported results, for the first time show the presence of a continuous layer of tetragonal zirconia phase developed in the proximity of pure zirconium substrate. Based on the Raman peak positions we prove that this tetragonal layer is stabilized by the high compressive stress and sub-stoichiometry level. Presence of the tetragonal phase located in the outer part of the scale have been confirmed, yet its Raman characteristics suggest a stress-free tetragonal phase, therefore different type of stabilization mechanism. Presented study suggest that its stabilization could be related to the lattice defects introduced by highstoichiometry of zirconia or presence of heterovalent cations.

I. INTRODUCTION

Due to their high corrosion resistance and almost complete transparency to neutrons, zirconium and its alloys are used as cladding elements in the nuclear industry. High temperature oxidation of zirconium has been extensively studied for many years. It has been shown that the high temperature oxidation of zirconium leads to the growth of an oxide scale which consists in a mixture of tetragonal and monoclinic phases [1-2]. It is commonly considered that the tetragonal phase promotes the protective role of the scale but the reasons for its stabilization are still under debate. Studies performed by Bouvier *et al.* [3], Godlewski *et al.* [4] and Barberis *et al.* [5] points to the conclusion that there are three possible phenomena responsible for the tetragonal phase stabilization, i.e.: crystallite size, point defects and stress state generated during oxidation. Therefore, it has been demonstrated that:

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